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Addendum to

Final

Report   January 1985

Evaluation and Prediction of Long-Term Environmental Effects on Nonmetallic Materials

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FOREWORD

This is an addendum to the final report for the third phase of the program, Evaluation and Prediction of Long-Term Space Environmental Effects on Nonmetallic Materials, conducted by Martin Marietta for the National Aeronautics and Space Administration, Marshall Flight Center, under Contract NAS8-33578.

The program was conducted in the Mechanical Materials Engineering Section with Mohan Misra as Program Manager and Harold Papazian as Principal Investigator.

Don Wilkes, of NASA-MSFC, served as Program Monitor. Dr. Ray Gause and Ms. Ann Whitaker served as Technical Advisors.

CONTENTS

INTRODUCTION

This is an addendum to the Final Report, January 1985 under Contract NAS8-33578. It is a result of changing Task 4 "Predictive Modeling" to encompass a somewhat broader range of environmental conditions and materials. It discusses: the in-flight data of the atomic oxygen reaction with carbon and osmium; the laboratory and in-flight data of the atomic oxygen reaction with polymeric films and the effect of electron irradiation on the rates of oxidation. No information has been found that could be used for modeling such effects on composites. Although not specifically required in Task 4, the effects of the space environment on thermal control coatings has been included because of its intrinsic interest, especially for Space Station.

Results

On STS-8, the TCQM was used to measure the quantitative oxidation of carbon and osmium films (Ref. 1). For the carbon loss a 2500A film was deposited on the TCQM, and the osmium film was 300A thick. Figure 1 shows a linear loss (i.e., constant with time) for the carbon film. Figure 2 shows the loss of the osmium film. In contrast with carbon, the osmium loss appears to take place in two steps, neither of which are linear with time. Data extracted from Figure 2 is presented in Figure 3 showing an exponential removal for both steps of the osmium loss.

The kinetics of hetrogeneous reactions, as studied in the laboratory, (Ref. 2) may be used to discuss such results. In a system consisting of a solid surface and a gas striking it, as a general rule, the latter will
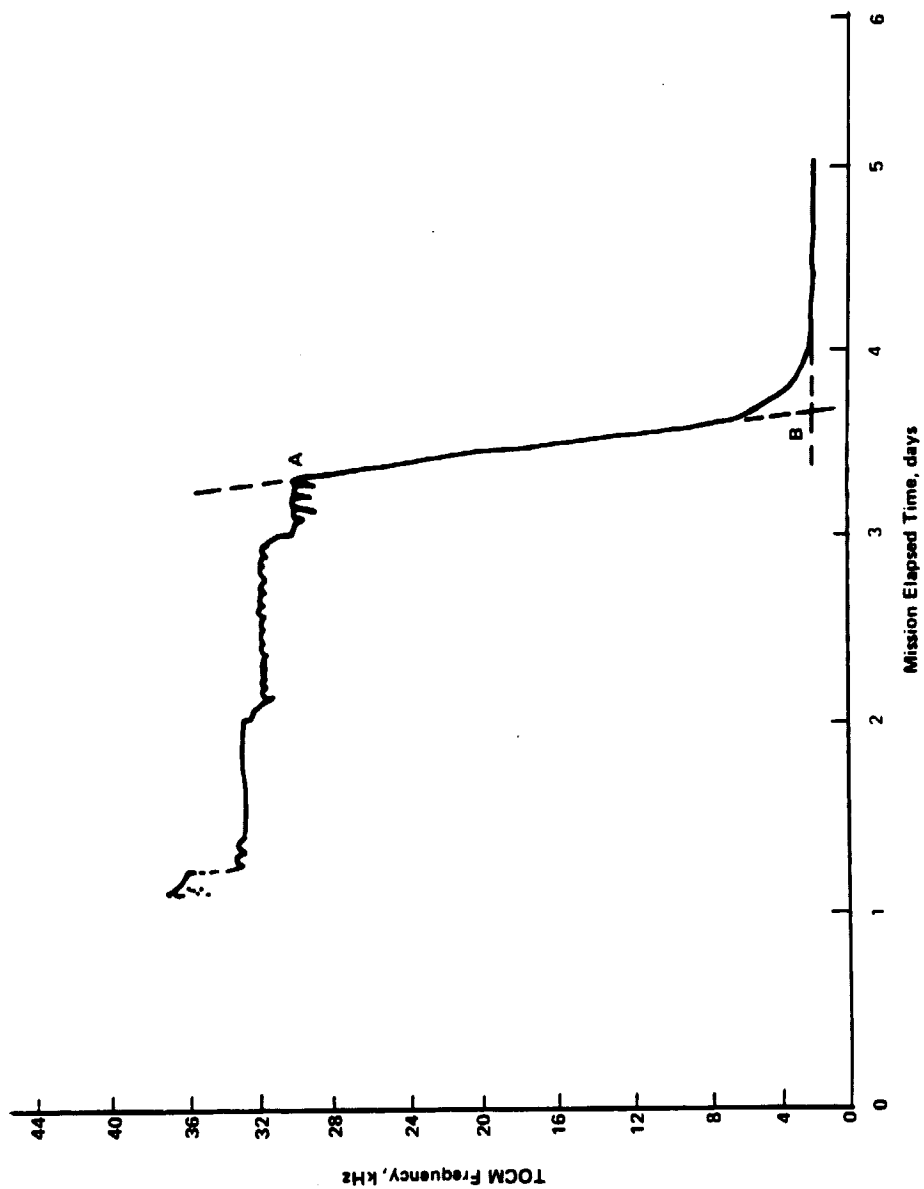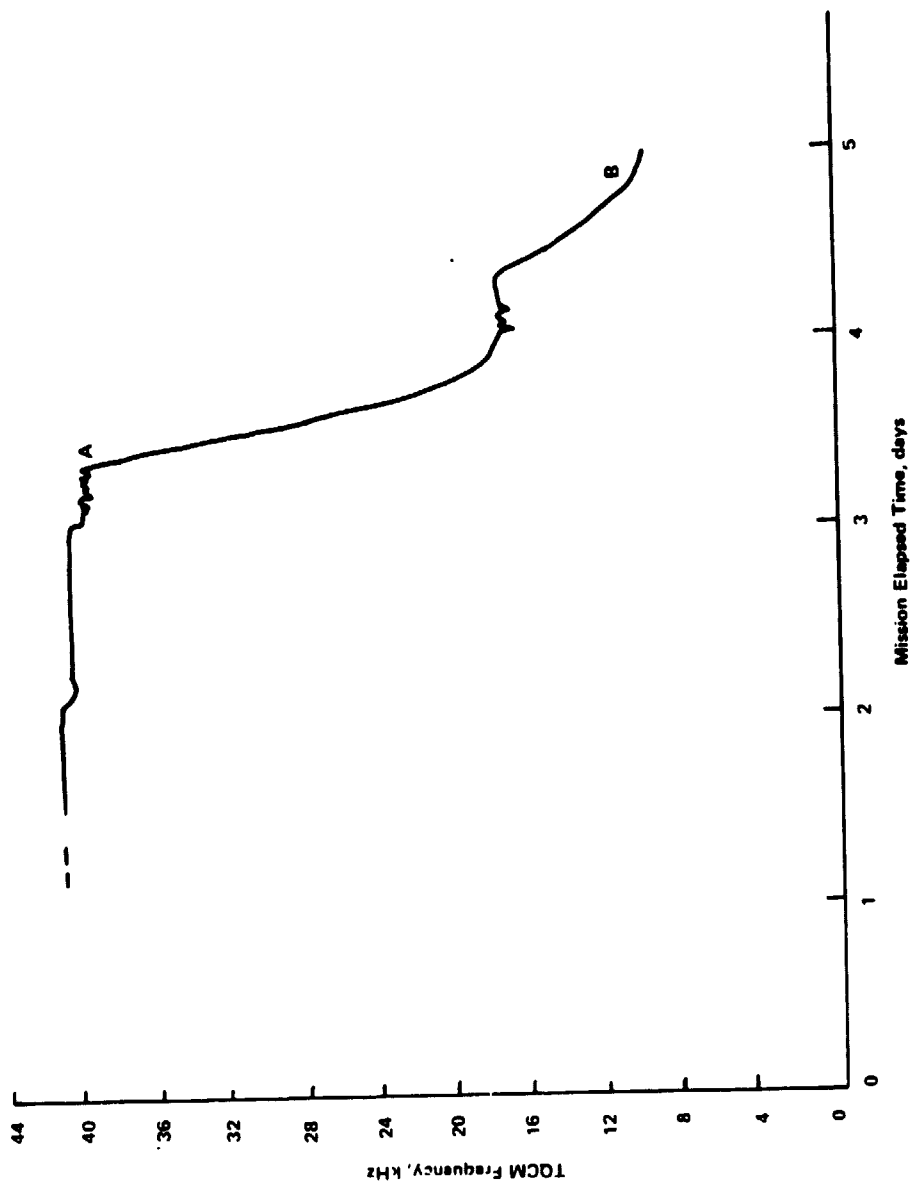
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Figure 1
STS-8 Atomic Oxygen Monitor TQCM 2—Carbon Coated (Facing out of Bay) (Ref 1)

Figure 2
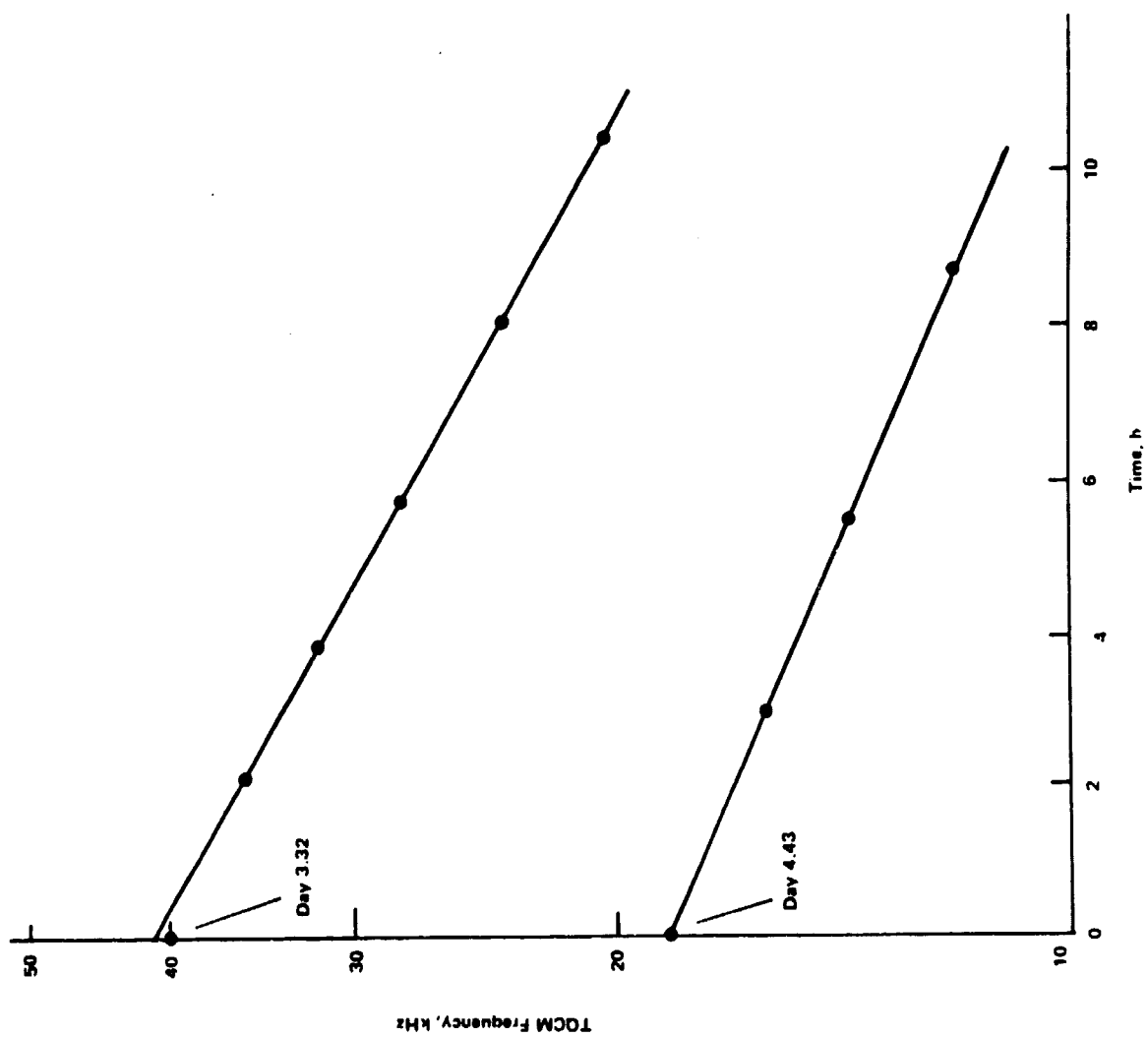*STS-8 Atomic Oxygen Monitor TQCM 4—Osmium Coated (Facing out of Bay) (Ref 1)*

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Figure 3 Kinetics Analysis of TQCM Data of Figure 2

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"condense" for a period of time. Then, as a result of thermal agitation, "evaporation" will take place from time to time. If α is the fraction of the gas which adheres (sticking coefficient), then ζ is the number which "condense" on each sq. cm of available surface per second, where is the number of striking one cm^2 per second. If θ is the fraction of the total surface covered with gas at any instant, then $1-\theta$ is the fraction of uncovered surface. Assuming that only a single layer of gas can form on the surface, the rate of condensation will be $(1-\theta)\alpha\zeta$ cm^{-2} sec^{-1}. The rate of "evaporation" will be proportional to the area covered, so that it may be represented by $\nu\theta$ where ν is a constant for the gas-surface system. When the rates of condensation and evaporation are equal,

$$(1-\theta)\alpha\zeta = \nu\theta \tag{1}$$

$$\theta = \alpha\zeta/(\alpha\zeta + \nu) \tag{2}$$

In general, chemical reaction may be considered to occur between m adjacent molecules, followed by evaporation of products, so that the rate of reaction is given by

$$dx/dt = \nu_1\theta^m \tag{3}$$

where $_1$ determines the rate of evaporation of the products and being a measure of the surface concentration of the reacting molecules. In the simplest case, m is equal to unity so that

$$dx/dt = \nu_1\theta \tag{4}$$

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Substituting the value of θ from Equation (2)

$$dx/dt = k_1 \zeta /(k_2 \zeta + 1) \tag{5}$$

where k_1 and k_2 are constants proportional to $\nu_1 \alpha /\nu$ and α/ν, respectively. With the assumption made above that only one gas molecule (or atom) is involved in the reaction, the process is unimolecular, but examination of Equation (5) shows that it is not a simple "first order" reaction (chemical kinetics terminology). However, in certain cases simplifications of Equation (5) are possible.

If the surface is sparsely covered (i.e., α is small), but evaporation is rapid (i.e., ν is large), θ will be small in comparison with unity; Equation (1) becomes $\alpha \zeta = \nu \theta$ and $k_2 = \alpha/\nu \rightarrow 0$, then Equation (5) yields

$$dx/dt = k_1 \zeta = k \zeta = kP \tag{6}$$

since ζ (collision frequency) is proportional to the pressure, P. The rate constant, k, has units of $time^{-1}$.

Thus a unimolecular heterogeneous reaction becomes kinetically first order when the surface is sparsely covered with molecules. Chemical reactions of the first order behave with time as depicted in Figure 3, and it appears that the loss of osmium can be described in this manner for both steps (Figure 2). However, this simplicity may not actually be the case for osmium, as the discussion below will indicate.

When the surface is almost completely covered ($\theta \rightarrow 1$), Equation (2) becomes
$\alpha \zeta = \alpha \zeta + \nu$, and Equation (5) simplifies to

$$dx/dt = k_1 \zeta/(k_2 \zeta + 1) = (\nu_1 \alpha \zeta)/(\alpha \zeta + \nu) = \nu_1$$

$$dx/dt = \text{constant} = k \qquad\qquad\qquad (7)$$

since ν_1 is constant, and the reaction is kinetically of "zero order"
(linear in time). The rate constant, k, for atom loss has units of atoms/time.

A. Oxidation of Carbon and Osmium

Figure 1 shows the carbon loss in flight to be linear for over 90% of the film
thickness. Thus, the oxidation is "zero order" kinetically. From the above
simplifications of Equation (5), it would appear that the reaction is
described by Equation (7). However, this requires the surface to be almost
completely covered with the reacting molecules. Almost certainly the surface
is sparsely covered, since the product of reaction is expected to be CO which
has a very high vapor pressure. Sparsely covered surfaces are described by
Equation (6) which is for a "first order" reaction in contrast to the observed
zero order. In the space environment, however, the collision frequency of
oxygen atoms with the surface is constant and the right-hand-side of Equation
(6), therefore, becomes constant. Thus, in space, the first order reaction
with sparsely covered surfaces is reduced to a zero order because of the
constancy of the collision frequency.

Just as a unimolecular reaction in the laboratory becomes of zero order when one of the products is firmly held on the surface (Equation 7), so a bimolecular process may, for the same reason, prove to be kinetically of the first order. Without question the osmium loss in flight is more complex than the carbon loss (compare Figures 1 and 2),and the analysis of Figure 3 shows it to be kinetically of first order; this however, does not preclude a bimolecular reaction (two oxygen atoms). If the osmium is removed as OsO, then as in the discussion for carbon, zero order kinetics would be expected. However, first order kinetics are observed. This implies that a bimolecular reaction (two oxygen atoms) may be occurring, and the reaction appears kinetically as first order. That this may well be the case is discussed below after introduction of the Leger reaction efficiency.

To account for thickness loss of materials, Leger defined the Reaction Efficiency (Ref. 3). It is derived by normalizing the thickness loss (or surface recession) by the oxygen atom fluence to yield $R.E = x \ cm^3$/oxygen atom. Implicit in this definition is the assumption of zero order kinetics since it defines the thickness loss as linear in time since the fluence contains time, or $\Delta thickness/\Delta time = $ constant.

Clearly for films where the thickness loss is complete, such as for carbon and osmium, the Leger R.E. can be in error. For example, on STS-8 the fluence is determined as 3.5×10^{20} oxygen atoms cm^{-2} from the 41 hours (1.5×10^5 secs) in the RAM direction but the carbon was lost in only 3×10^4 sec (between points A and B in Figure 1) and the osmium in 10.1×10^4 sec (between points A and B in Figure 2). The Leger R.E. for carbon is 0.71×10^{-25} and for osmium it is $.086 \times 10^{-25}$, whereas when corrected for time, the reaction efficiency is some 5 times larger for carbon and 1.5 times larger for

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osmium. It should be noted that even this is not correct for osmium since the osmium kinetics are not zero order but rather first order for both steps in the loss (see Figure 3).

The rate constant for the zero order kinetics of carbon is easily obtained. The area of the 2500A thick sample was 5.06 cm^2 for a total volume of carbon 12.65×10^{-5} cm^3. From the density of graphite (2.267 gm cm^{-3}) and the molar volume = 5.3 cm^3, therefore, the total number of carbon atoms lost is $(12.65 \times 10^{-5}/V_m)$ 6.02×10^{23} = 14×10^{18} atoms. Thus, the rate constant, k, becomes 14×10^{18} atoms/3×10^4 sec = 4.4×10^{14} carbon atoms/sec. The number of oxygen atoms required to remove a carbon atom is given by:
flux/rate constant = $2.32 \times 10^{15}/4.4 \times 10^{14}$ = 5.2.

Alternatively, a reaction probability, P, can be defined as P = R.E. x (area of loss)/volume of carbon atom = .207 and then 1/P = 4.8, in good agreement with that obtained from the rate constant. This is, of course, essentially equal to: correct fluence/number of carbon atoms = $7 \times 10^{19}/1.4 \times 10^{19}$ = 5. The latter is the simplest method for determining an efficiency defined as the number of oxygen atoms to remove one carbon atom.

For the osmium loss there appears to be two separate first order reactions (see Figure 3). The rate constants may be calculated from the half life, $t_{1/2}$, for each reaction. In the first step, half of the thickness is lost in 1.8×10^4 seconds so that the rate constant is k = ln $2/t_{1/2}$ = 3.8×10^{-5} sec $^{-1}$. For the second step, half of the remaining 72A is lost in 1.7×10^4 sec so that k = 4×10^{-5} sec $^{-1}$. The closeness of those values may indicate some malfunction of the quartz microbalance for a time, near day 4 (Figure 2), with subsequent recovery.

The first order kinetics equation (Equation 6) can be written for the loss of osmium as

$$dx/dt = k(a_0-x) \tag{8}$$

where a_0 is the original amount of osmium and x is the amount lost so that a_0-x is the amount remaining at time t. Equation 8 can be integrated to yield

$$a_0-x = a_0 \exp(-kt)$$

or $\quad (a_0-x)/a_0 = \exp(-kt) = $ fraction remaining

so that $\quad 1- \exp(-kt) = $ fraction lost at time t.

For example, to calculate the time to 99% loss for the first reaction

$$1- \exp(-3.8 \times 10^{-5}t) = .99$$

$$\exp(-3.8 \times 10^{-5}t) = 1-.99 = .01$$

$$-3.8 \times 10^{-5}t = -4.61$$

or $\quad t = 4.61/3.8 \times 10^{-5} = 1.21 \times 10^5 $ sec.

For the second reaction the time to 99% loss of the remaining 72A of film is

$$-4 \times 10^{-5}t = -4.61$$

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or $\qquad$ t = -4.61/4x10^5 = 1.15x10^5 sec.

The similarity in the times is a result of the similarity in the rate constants, assuming no malfunction of the quartz microbalance.

If there was no malfunction of the quartz microbalance during loss of the osmium, then the reaction efficiency can be instructive. For the first step, the fluence (with t=1.21x10^5 sec) is calculated as 2.8x10^{20} oxygen atoms cm^{-2}. The total number of osmium atoms is calculated as 8.2x10^{17} (for 228A); therefore, 2.8x10^{20}/8.2x10^{17} = 341 oxygen atoms are required to remove one osmium atom. In the second step 4.2x10^4 seconds are required to remove the final 72A. The fluence is calculated as 2.7x10^{20} oxygen atoms cm^{-2} and the total number of osmium atoms removed is 2.6x10^{17} or 2.7x10^{20}/2.6x10^7 = 1004 oxygen atoms to remove one osmium atom. From the ratio 1004/341 = 2.9 it may be assumed that three times as many oxygen atoms are required in the second step compared to the first step. If it is assumed that in the first step the osmium is lost as OsO_2, then the osmium loss in the second step is obviously more inefficient and probably involves OsO_4. The reason for the change in the chemistry (if real) is unclear.

B. Oxidation of Polymeric Films

Table 1 shows the results of flight data on polymeric films from several STS flights. Figure 4 shows laboratory results of the effects of atomic oxygen on various polymers (Ref. 4). The figure shows the reactions to be linear in time, i.e., zero order. Thus, the implicit assumption of zero order

Table 1 Atomic Oxygen Reaction Efficiencies

Shuttle Flight	Material	Fluence, 10^{20} Atoms/cm^2	Reaction Efficiency, cm^3/Atom x 10^{24}
STS-3	Kapton TV Blanket	2.16	2.0
	Kapton, OSS-1 Blanket		2.5
STS-4	Kapton MLI Blanket		
	Kapton	0.65	2.8
	Kapton		2.7
	Kapton		2.6
	Mylar		2.8
	Teflon FEP 7 TFE		0.1
	Al/Teflon FEP		
STS-5	Kapton	1.0	1.5
	Kapton		2.2
	Kapton		2.8
	Kapton, Black		1.4
	Mylar		2.2
	Mylar		1.8
	Mylar		1.5
	Tedlar, Clear		1.3
	Tedlar, White		0.4
	Teflon, FEP & TFE		0.2
	Kapton (Coated)		
	DC1-2755		0.2
	T-650		0.2
STS-8	Kapton	3.5	
	Kapton		3.0
	Kapton		
	Mylar A		3.6
	Mylar A		3.4
	Mylar D		3.0
	Clear Tedlar		3.2
	Polyethylene		3.3
	Teflon TFE		<0.05
	Kapton F		<0.05

Notes:
1. Polysulfide Polymer.
2. Formaldehyde Ploymer.
3. Polypropylene.
4. Low-density Polyethylene.
5. Polyethylene Glycol Terephthalate
6. Polystyrene.
7. Polytetraflouroethylene.
8. Sulfur-Vulcanized Natural Rubber

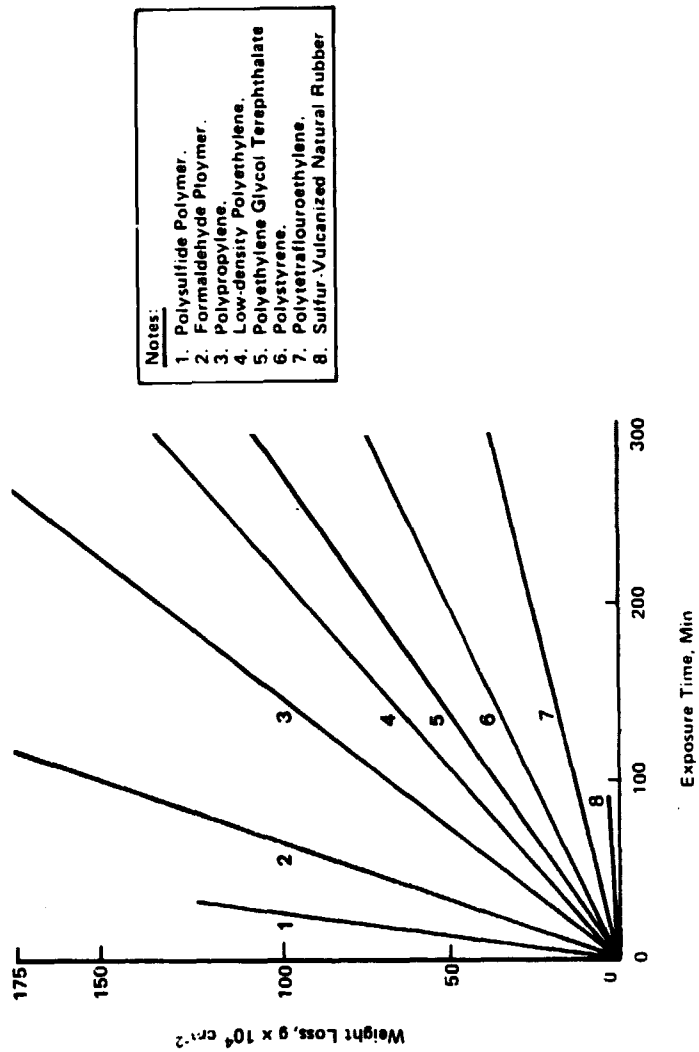Weight Loss, g x 10^4 cm.$^{-2}$

Exposure Time, Min

Figure 4 Relative Effects of Atomic Oxygen on a Variety of Polymers (Ref 4)

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kinetics in the Leger R.E. appears to be correct. Since none of the polymeric films disappeared completely in flight, the fluence as calculated from the time in the RAM direction may be used in the calculations of R.E.

Table 2 shows the weight loss $cm^{-2}sec^{-2}$ for a variety of polymers in the laboratory study (Ref. 4). The oxygen atom concentrations were not quantitatively measured but were estimated to be in the range 10^{-14}-10^{-15} atoms cm^{-3}, at a pressure of 1 mm flowing over the sample at 4 cm^3 min^{-1}. Flowing of the gas ensures the collision frequency to remain essentially constant as in the space environment. From Table 2 the values of polyimide and polyethylene terephthalate may be compared with the flight specimens Kapton and Mylar, respectively.

The total collision frequency, ζ , for the laboratory studies may be calculated as (Ref. 2)

$$\zeta = (3.5 \times 10^{22}/(MT)^{1/2})P_{mm} = 2.5 \times 10^{20} \text{ collisions } cm^{-2} \text{ } sec^{-1}$$

where P = 1 mm, T = 300^{0}K, and M = 32 since the oxygen is predominantly as molecules. The number of collisions of oxygen atoms (10^{14} cm^{-3}) may be estimated from the number of total molecules at 1 atm. at 300^{0}K which is about 2.4×10^{19} cm^{-3} then

$$1 \times 10^{14}/2.4 \times 10^{19} = 4.1 \times 10^{-6} \text{ atm } \times 760 \text{ mm/atm} = 3.1 \times 10^{-3} \text{ mm}$$

and the collision frequency for the oxygen atoms becomes

$$(2.5 \times 10^{20})(3.1 \times 10^{-3}) = 7.8 \times 10^{17} \text{ collisions } cm^{-2} \text{ } sec^{-1}.$$

Table 2 Atomic Oxygen Reaction with Polymers (Ref 4)

Type of Polymer	$\varrho \times 10^7$ cm^{-2} s^{-1}
Low-Density Polyethylene	8.55
Irradiated Low-Density Polyethylene (1 Mrad)	9.55
Irradiated Low-Density Polyethylene (10 Mrad)	11.76
Irradiated Low-Density Polyethylene (105 Mrad)	14.21
Chemically Crosslinked Low-Density Polyethylene	11.41
Low Molecular Weight Highly Branched Polyethylene	11.12
High-Density Ethylene-Butene Copolymer	10.66
Polypropylene	11.93
Polybutene-1	12.28
Chlorinated High-Density Polyethylene	17.24
Chlorinated Polyethylene plus 10% Polysulfide Polymer	10.00
Natural Rubber	11.69
Natural Rubber—Sulfur Raw Stock	4.14
Natural Rubber—Sulfur Vulcanizate	0.55
Natural Rubber—Peroxide Raw Stock	10.31
Natural Rubber—Peroxide Cured	5.76
Commercial Hard Rubber	9.34
Vulcanized Ethylene-Propylene Rubber	0.67
Polystyrene	4.23
Poly-3-Phenyl-1-Propene	4.93
Poly-4-Phenyl-1-Butene	5.76
Polyvinylenecyclohexane	7.86
ABS Polymers, Several Types	9.24
Unplasticized Polyvinyl Chloride Copolymer	16.24
Polyvinyl Fluoride	8.76
Polytetrafluoroethylene	2.14
Perfluorinated Ethylene-Propylene Copolymer	1.52
Polymethyl Methacrylate	7.38
Polyimide	4.10
Polycarbonate	8.93
Polyethylene Terephthalate	6.28
Nylon G	9.55
Nylon 610	11.17
Formaldehyde Polymers	19.90—27.10
Polysulfide (Chloroethyl Formal Disulfide)	67.1
Cellulose Acetate	17.24

From Table 2 for polymimide (Kapton) the weight loss is 4.1×10^{-7} gm cm^{-2} sec^{-1} then $4.1 \times 10^{-7}/7.8 \times 10^{17} = .5 \times 10^{-24}$ gm/oxygen atom. For Mylar it is $6.28 \times 10^{-7}/7.8 \times 10^{17} = .8 \times 10^{-24}$ gm/oxygen atom.
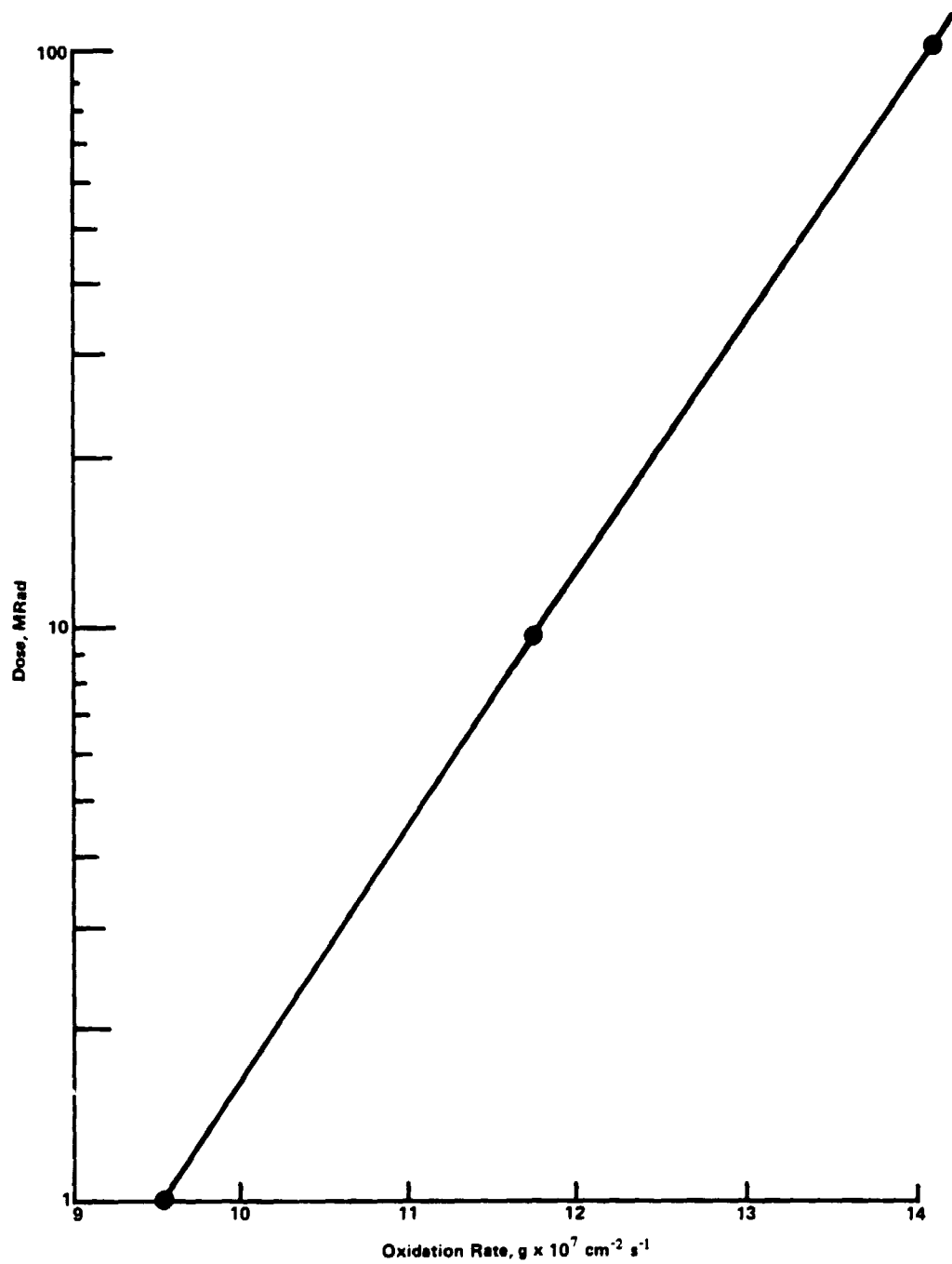
Table 1 shows the flight R.E. for Kapton to range from $(2-3) \times 10^{24}$ cm^3/oxygen atom taking the density of Kapton to be 1.4 gm cm^{-3}, this is equivalent to $(2.8-4.2) \times 10^{-24}$ gm/oxygen atom. For Mylar the R.E. ranges from $(1.5-3.6)10^{-24}$ and with a density of 1.37, this is equivalent to $(2-4.9) \times 10^{-24}$ gm/oxygen atom. The flight results for Kapton are about a factor of 4-6 greater than observed in the laboratory tests and about 2.5-6 times greater for Mylar. If the actual oxygen atom concentration was somewhat less than the estimated 10^{14} cm^{-3} in the laboratory tests, the agreement would be excellent. It appears that laboratory studies can give correct results (if the fluence in flight and concentration of oxygen atoms in the laboratory are known accurately).

Table 2 contains information of particular importance for the space environment. It can be seen that polyethylene oxidation is enhanced by electron irradiation (energy not specified). The results have been plotted in Figure 5 from which the rate of oxidation as a function of dose can be obtained as

$$\log (\text{MRads}) = \text{slope}(\text{Rate}) + \log \text{constant}$$

or

$$\text{Rate} = \frac{1}{\text{slope}} \log \left(\frac{\text{MRads}}{\text{const}} \right)$$

Figure 5 Oxidation Rate of Polyethylene as a Function of Electron Irradiation

which for polyethylene is

$$Rate = 2.3 \times 10^{-7} \log\left(\frac{MRads}{6 \times 10^{-5}}\right)$$

Further laboratory testing of other polymer oxidation rates as a function of dose is very much warranted.

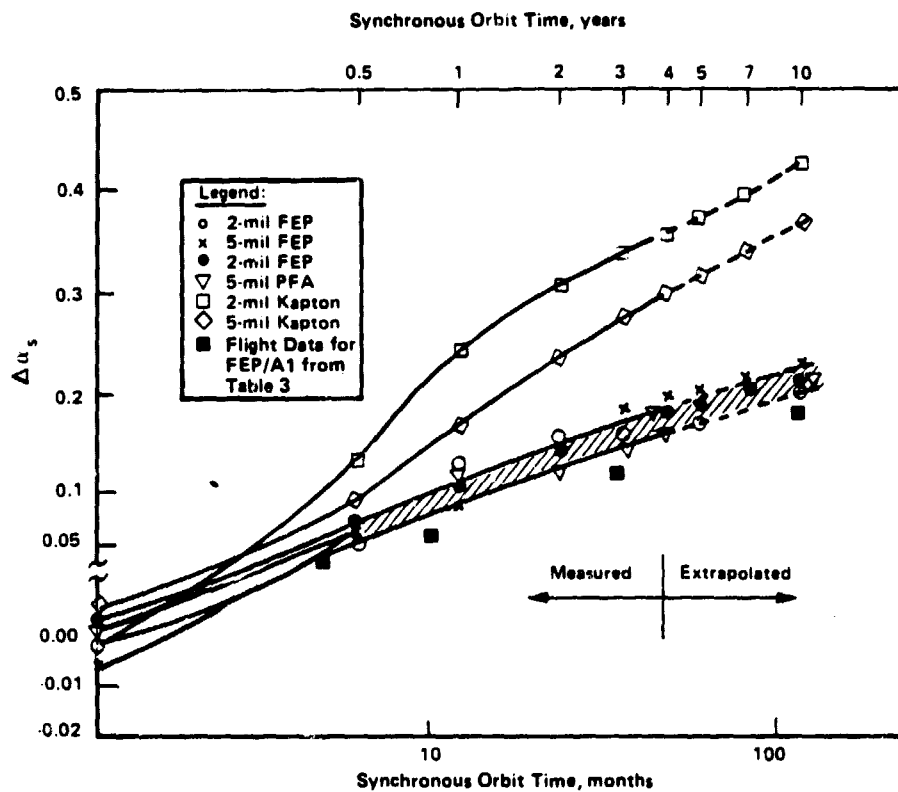C. Environmental Effects on Thermal Control Coatings

The AFML experiment M1-101 was launched on the STP P72-1 satellite in October of 1972 (Ref. 5). The experiment studied changes in solar absorptance of thermal control coatings at 400 nautical miles. Data were telemetered for 5 years. Similar coatings were flown in Skylab D024 (Ref. 6). The results from both flights were analyzed and compared. The analysis showed that while Skylab changes were caused by contamination, the changes in the ML-101 experiment were caused by the natural environment and not by contamination (Ref. 7). The results of the analysis are listed in Table 3 along with the predicted values of $\Delta\alpha_s$ after 10 years.

The flight results for FEP/Al from Table 3 have been plotted on Figure 6 which shows laboratory results on the combined effects of u.v., protons and electrons on metalized films of FEP and Kapton to simulate an environment at synchronous orbit (22000 nautical miles). (Figure 6 was developed for the Final Report, May 1983 (Ref. 8)). At 400 nautical miles the estimated flux for electrons (energy $\geq$ .25MeV) is about 25% of that at 400 nautical miles, while the estimated flux for protons (energy $\geq$ .1MeV) is about 0.4% so that the

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Table 3
Thermal Control Coating Changes in the Natural Environment at 400 nmi

Thermal Control Coating	Observed $\Delta\alpha_s$ Changes[a]	Time Period, months[a]	$\Delta\alpha_s$	x→120[b]	Predicted $\Delta\alpha_s$ after 10 years in Flight
S-13G	$= 0.035t^{.58}$	0-16	0.17		
	$= 0.08t^{.26}$	16-60		0.27	0.44
Zn_2TiO_4	$= 0.03t^{.58}$	0-17	0.16		
	$= 0.09t^{.26}$	17-60		0.30	0.46
TiO_2	$= 0.024t^{.7}$	0-20	0.20		
	$= 0.06t^{.36}$	20-60		0.31	0.51
Al_2O_3	$= 0.05t^{.65}$	0-5	0.14		
	$= 0.07t^{.47}$	5-20	0.25		
	$= 0.15t^{.19}$	20-60		0.36	0.75
Eu_2O_3	$= 0.02t^{.65}$	0-20	0.14		
	$= 0.044t^{.34}$	20-60		0.21	0.35
SiO_2 (Fabric)	$= 0.028t^{.46}$	0-12	0.09		
	$= 0.05t^{.22}$	12-60		0.14	0.23
FEP/Al	$= 0.017t^{.5}$	0-10	0.06		
	$= 0.03t^{.16}$	10-36	0.05		
	$= 0.0046t^{.68}$	36-60		0.09	0.19
OSR	$= 0.014t^{.5}$	0-10	0.04		
	$= \sim 0$	10-36	0		
	$= 0.013t^{.39}$	36-60		0.07	0.11

Note:
a. From Ref 7.
b. SG-13, X=16, FEP/A1, X=36; etc.

Figure 6
Changes in Solar Absorptance Characteristics of Metalized Films of Teflon
and Kapton As a Function of Time (Radiation Near-UV and Far-UV,
27-keV Protons, 7, 80, and 200-keV Electrons; Points Are Average of
Three Samples Each)

flight data should show less changes than those expected in synchronous orbit. Thus, the flight data and laboratory data (cross-hatched curve) comparisons in Figure 6 are gratifyingly good.

Conclusions

The effects of atomic oxygen on polymeric films can be modeled in the laboratory. Electron irradiation enhances the rate of oxidation of polyethylene. Studies on other polymers should be carried out.

The changes in solar absorptance can be modeled in the laboratory.

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